CLAIMS

- 1. Method of manufacturing a metal compound such as metal oxides, metaloxy hydroxides metal hydroxides, metal carbides, metal carbides, metal borides,
- 5 electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the steps of:
 - introducing a solid reactor filling material in a reactor,
 - introducing a metal-containing precursor in said reactor,
 - introducing a reactant into said reactor,
- 10 introducing a supercritical solvent into the said reactor, thereby
 - establishing a contact between the metal-containing precursor and the co-solvent, thus
 - resulting in the formation of said compound in the proximity of the said solid reactor filling material.
- 2. Method of manufacturing a semi-metal compound such as semi-metal oxides, semi-metaloxy hydroxides semi-metal hydroxides, semi-metal carbides, semi-metal nitrides, semi-metal carbonitrides, semi-metal borides, electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the steps of:
 - introducing a solid reactor filling material in a reactor,
- 20 introducing a semi-metal-containing precursor in said reactor,
 - introducing a reactant into said reactor,
 - introducing a supercritical solvent into the said reactor, thereby
 - establishing a contact between the semi-metal-containing precursor and the co-solvent, thus
- resulting in the formation of said compound in the proximity of the said solid reactor filling material.
 - 3. Method of manufacturing a metal compound such as metal oxides, metaloxy hydroxides metal hydroxides, metal carbides, metal nitrides, metal carbonitrides, metal borides,
- 30 electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the steps of:
 - introducing a solid reactor filling material in a reactor,
 - introducing a metal-containing oxide in said reactor,
 - introducing a substitution source into said reactor,
- 35 introducing a supercritical solvent into the said reactor, thereby
 - establishing a contact between the metal-containing oxide and the substitution source, thus

42

PCT/DK2003/000934

- resulting in the formation of said compound in the proximity of the said solid reactor filling material.
- 4. Method of manufacturing a semi-metal compound such as semi-metal oxides, semi-5 metaloxy hydroxides semi-metal hydroxides, semi-metal carbides, semi-metal nitrides, semi-metal carbonitrides, semi-metal borides, electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the steps of:
 - introducing a solid reactor filling material in a reactor,
 - introducing a semi-metal-containing oxide in said reactor,
- 10 introducing a substitution source into said reactor,

WO 2005/061410

- introducing a supercritical solvent into the said reactor, thereby
- establishing a contact between the semi-metal-containing oxide and the substitution source, thus
- resulting in the formation of said compound in the proximity of the said solid reactor filling material.
 - 5. Method according to any of claims 1-4, said method also comprising the step of introducing a co-solvent into said reactor.
- 20 6. Method according to any of claims 1-5, wherein the formation of said compound takes place by a process involving at least a sol-gel reaction.
 - 7. Method according to claim 1 or claim 3, wherein the metal compound is substantially crystalline.
 - 8. Method according to claim 2 or claim 4, wherein the semi-metal compound is substantially crystalline.
- Method according to claim 1 or claim 3, wherein the metal compound is substantially
 amorphous.
 - 10. Method according to claim 2 or claim 4, wherein the semi-metal compound is substantially amorphous.
- 35 11. Method according to claim 1 or claim 3, wherein the metal compound is a mixture of several different phases.
 - 12. Method according to claim 2 or claim 4, wherein the semi-metal compound is a mixture of several different phases.

43

PCT/DK2003/000934

WO 2005/061410

- 13. Method according to any of claims 1 or 3 and any of claims 5-12, wherein the introduction of the solid reactor filling material, the metal-containing precursor, alternatively the semi-metal precursor, the possible co-solvent, and the supercritical
 5 solvent into the said reactor is done in arbitrary order.
- 14. Method according to any of claims 2 or 4 and any of claims 5-12, wherein the introduction of the solid reactor filling material, the metal-containing oxide, alternatively the semi-metal-containing oxide, the possible co-solvent, and the supercritical solvent into
 10 the said reactor is done in arbitrary order.
- 15. Method according to any of claims 1 or 3 and any of claims 5-14, wherein at least one of the solid reactor filling material, the metal-containing precursor, alternatively the semi-metal-containing precursor, the possible co-solvent or the supercritical solvent is mixed
 15 with at least one of the solid reactor filling material, the metal-containing precursor, alternatively the semi-metal-containing precursor, the possible co-solvent or the supercritical solvent before introduction into the said reactor.
- 16. Method according to any of claims 2 or 4 and any of claims 5-14, wherein at least one of the solid reactor filling material, the metal-containing compound, alternatively the semi-metal-containing compound, the possible co-solvent or the supercritical solvent is mixed with at least one of the solid reactor filling material, the metal-containing compound, alternatively the semi-metal-containing compound, the possible co-solvent or the supercritical solvent before introduction into the said reactor.
 - 17. Method according to any of claims 1 or 3 and any of claims 5-16, where the reactant comprises at least one of the following components: water, ethanol, methanol, hydrogenperoxid and isopropanol.
- 30 18. Method according to any of claims 2 or 4 and any of claims 5-17, where the substitution source comprises at least one of the following components: carbon, nitrogen, boron and/or any combination of these.
- 19. Method according to any of claims 1 or 3 and any of claims 5-18, wherein the metal35 compound is manufactured in a mode selected from the group of: a batch mode, a quasi-batch mode and a substantially continuos mode.

20. Method according to any of claims 2 or 4 and any of claims 5-18, wherein the semimetal compound is manufactured in a mode selected from the group of: a batch mode, a quasi-batch mode and a substantially continuos mode.

44

PCT/DK2003/000934

WO 2005/061410

10

- 5 21. Method according to any of claims 1-20, wherein a temperature in the reactor during the formation of said compound is kept at a fixed temperature.
 - 22. Method according to any of claims 1-20, wherein a temperature in the reactor during the formation of said compound is performed at an increasing temperature.

23. Method according to any of claims 1-20, wherein a temperature in the reactor during the formation of said compound is performed at a decreasing temperature.

24. Method according to any of claims 1-20, wherein a temperature in the reactor during the formation of said compound is performed at a temperature profile being an arbitrary combination at least two of the temperature profiles: a fixed temperature, an increasing temperature, a decreasing temperature.

- 25. Method according to any of claims 21-24, wherein the temperature in the reactor during the formation of said compound is maximum 400°C, more preferably maximum 300°C, even more preferably maximum 200°C, most preferably maximum 100°C, and even and most preferably maximum 50°C.
- 26. Method according to any of claims 1-25, wherein a pressure in the reactor during the formation of said compound is kept at a fixed pressure.
 - 27. Method according to any of claims 1-25, wherein a pressure in the reactor during the formation of said compound is performed at an increasing pressure.
- 30 28. Method according to any of claims 1-25, wherein a pressure in the reactor during the formation of said compound is performed at a decreasing pressure.
- 29. Method according to any of claims 26-28, wherein a pressure in the reactor during the formation of said compound is performed at a pressure profile being an arbitrary35 combination at least two of the pressure profiles: a fixed pressure, an increasing pressure, a decreasing pressure.

30. Method according to any of claims 1-29, wherein the supercritical solvent is CO_2 , and wherein the pressure in the reactor during the formation of said compound is minimum 74 bar, more alternatively minimum 80 bar, even more alternatively minimum 90 bar, and most alternatively minimum 100 bar.

45

PCT/DK2003/000934

5

WO 2005/061410

- 31. Method according to any of claims 1-30, wherein the supercritical solvent is CO₂, and wherein the temperature in the reactor during the formation of said compound is minimum 31°C, alternatively 43°C, alternatively minimum 100°C, alternatively minimum 200°C, alternatively minimum 300°C, alternatively minimum 400°C alternatively minimum 500°C, alternatively minimum 600°C, alternatively minimum 700°C, alternatively minimum 800°C.
- 32. Method according to any of claims 1-29, wherein the supercritical solvent is isopropanol, and wherein the pressure in the reactor during the formation of said compound is minimum 47 bar, more alternatively minimum 80 bar, even more alternatively minimum 90 bar, and most alternatively minimum 100 bar.
- 33. Method according to any of claima 1-29 or claim 32, wherein the supercritical solvent is isopropanol, and wherein the temperature in the reactor during the formation of said compound is minimum 235°C, more alternatively minimum 250°C, even more alternatively minimum 270°C, most alternatively minimum 300°C, and even and most alternatively minimum 400°C.
 - 34. Method according to any of claims 1-33, wherein the supercritical solvent is supercritical before the introduction into said reactor.

- 35. Method according to any of claims 1-33, wherein the supercritical solvent is brought into a supercritical phase after the introduction into said reactor.
- 36. Method according to any of claims 1-35, wherein the time of the formation of said compound is maximum 1 hour, preferably maximum 0,75 hour, and most preferably maximum 0,5 hour.
- 37. Method according to any of claims 1-36, wherein the time of the formation of said compound is maximum 8 hours, preferably maximum 6 hours, and most preferably35 maximum 2 hours.
 - 38. Method according to any of claims 1-37, wherein the time of the formation of said compound is maximum 24 hours, preferably maximum 17 hours, and most preferably maximum 10 hours.

39. Method according to any of claims 1 or 3 and any of claims 5-38, wherein a plurality of

46

different metal-containing precursors is introduced in said reactor.

PCT/DK2003/000934

WO 2005/061410

10

25

- 5 40. Method according to any of claims 2 or 4 and any of claims 5-38, wherein a plurality of different semi-metal-containing precursors is introduced in said reactor.
 - 41. Method according to any of claims 1 or 3 and any of claim 5-38, wherein a plurality of different metal-containing oxides is introduced in said reactor.

42. Method according to any of claims 2 or 4 and any of claims 5-38, wherein a plurality of different semi-metal-containing oxides is introduced in said reactor.

- 43. Method according to any of claims 1 or 2 and any of claims 5-42, wherein the metalcontaining or semi-metal containing precursor is a metal alkoxide or a semi-metal alkoxide.
- 44. Method according to any of claims 1 or 3 and any of claims 5-43, wherein the metal-containing precursor or the metal containing oxide is selected from the group of: titanium 20 tetraisopropoxide, titanium butoxide, titanium ethoxide, and titanium methoxide.
 - 45. Method according to any of claims 1 or 3 and any of claims 5-43, wherein the metal-containing precursor or the metal-containing oxide is selected from the group of: aluminium isopropoxide and aluminium-sec-butoxide.

46. Method according to any of claims 1 or 3 and any of claims 5-43, wherein the metal-containing precursor or the metal-containing oxide is magnesium ethoxide.

- 47. Method according to any of claims 1 or 3 and any of claims 5-43, wherein the metal-30 containing precursor or the metal-containing oxide is a metal salt.
 - 48. Method according to any of claims 2 or 4 and any of claims 5-43, wherein the semimetal-containing precursor or the semi-metal-containing oxide is a semi-metal salt.
- 35 49. Method according to any of claims 1 or 3 and any of claims 5-43, wherein the metal-containing precursor or the metal-containing oxide is $Ti(SO_4)_2$.

WO 2005/061410 PCT/DK2003/000934

- 50. Method according to any of claims 1 or 3 and any of claims 5-43, wherein the metal-containing precursor or the metal-containing oxide is selected from the group of: $TiCl_4$ and $AlCl_3$.
- 5 51. Method according to any of claims 5-50, wherein the co-solvent is selected from the group of: water, ethanol, methanol, hydrogenperoxid and isopropanol.
 - 52. Method according to any of claims 5-51, wherein a plurality of different co-solvents is introduced in said reactor.

53. Method according to any of claims 1-52, wherein the solid reactor filling material functions as a heterogeneous catalyst.

10

25

- 54. Method according to claim 53, wherein the solid reactor filling material comprises at least one promoter.
 - 55. Method according to any of claims 1-54, wherein the solid reactor filling material is constituted by at least one fibre.
- 20 56. Method according to any of claims 1-54, wherein the solid reactor filling material is constituted by a powder.
 - 57. Method according to any of claims 1-54, wherein the solid reactor filling material has the shape selected from the group of: a sponge, a grid, a wad of fibres, and a sheet.
 - 58. Method according to any of claims 1-57, wherein the solid reactor filling material has a substantially porous structure.
- 59. Method according to any of claims 1-58, wherein the solid reactor filling material has a size and shape capable of substantially confining the metal-containing precursor to a limited part of the reactor.
 - 60. Method according to any of claims 1-59, wherein the solid reactor filling material comprises a polymer.
 - 61. Method according to claim 60, wherein the polymer is selected from the group of: polystyrene (PS), polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC), polyvinylidene chloride (PVDC), and polyvinyl acetate (PVAc).

48

WO 2005/061410

20

25

62. Method according to claim 60, wherein the polymer is selected from the group of: acrylic polymer, fluorinated polymer, diene polymer, vinyl copolymer, polyamide polymer, polyester polymer, polyether polymer, and polyimide polymer.

PCT/DK2003/000934

- 5 63. Method according to any of claims 1-62, wherein the solid reactor filling material comprises a metal.
- 64. Method according to claim 63, wherein the metal is selected from the group of: titanium, aluminium, zinc, vanadium, magnesium, zirconium, chromium, molybdenum,10 niobium, tungsten, copper, and iron.
 - 65. Method according to any of claims 1-62, wherein the solid reactor filling material comprises a semi-metal.
- 15 66. Method according to claim 65, wherein the metal is selected from the group of: silicon and boron.
 - 67. Method according to any of claims 1-64, wherein the solid reactor filling material comprises a metal compound.
 - 68. Method according to claim 67, wherein the metal compound is selected from the group of: titanium oxide, zinc oxide, copper oxide, aluminium oxide, vanadium oxide, magnesium oxide, zirconium oxide, chromium oxide, silicon oxide, molybdenum oxide, niobium oxide, tungsten oxide, hafnium oxide, tantalum oxide and iron oxide.
- 69. Method according to claim 67, wherein the metal compound is selected from the group of: titanium carbide, zinc carbide, copper carbide, aluminium carbide, vanadium carbide, magnesium carbide, zirconium carbide, chromium carbide, silicon carbide, molybdenum carbide, niobium carbide, tungsten carbide, hafnium carbide, tantalum carbide, cobalt
 30 carbide, manganese carbide, nickel carbide, berylium carbide and iron carbide.
- 70. Method according to claim 67, wherein the metal compound is selected from the group of: titanium nitride, zinc nitride, copper nitride, aluminium nitride, vanadium nitride, magnesium nitride, zirconium nitride, chromium nitride, silicon nitride, molybdenum
 35 nitride, niobium nitride, tungsten nitride, hafnium nitride, tantalum nitride, cobalt nitride, manganese nitride, nickel nitride, berylium nitride and iron nitride.

WO 2005/061410 PCT/DK2003/000934

- 71. Method according to claim 67, wherein the metal compound is selected from the group of: titanium carbonitride, zinc carbonitride, copper carbonitride, aluminium carbonitride, vanadium carbonitride, magnesium carbonitride, zirconium carbonitride, chromium carbonitride, silicon carbonitride, molybdenum carbonitride, niobium carbonitride, tungsten carbonitride, hafnium carbonitride, tantalum carbonitride, cobalt carbonitride, manganese carbonitride, nickel carbonitride, berylium carbonitride and iron carbonitride.
- 72. Method according to claim 67, wherein the metal compound is selected from the group of: titanium boride, zinc boride, copper boride, aluminium boride, vanadium boride,
 10 magnesium boride, zirconium boride, chromium boride, silicon boride, molybdenum boride, niobium boride, tungsten boride, hafnium boride, tantalum boride, cobalt boride, manganese boride, nickel boride, berylium boride and iron boride.
- 73. Method according to any of claims 1-64, wherein the solid reactor filling material comprises a semi-metal compound.
 - 74. Method according to claim 73, wherein the semi-metal compound is selected from the group of: silicon oxide and boron oxide.
- 20 75. Method according to claim 73, wherein the semi-metal compound is selected from the group of: silicon nitride and boron nitride.
 - 76. Method according to claim 73, wherein the semi-metal compound is selected from the group of: silicon carbonitride and boron carbonitride.

25

- 77. Method according to claim 73, wherein the semi-metal compound is selected from the group of: silicon boride and boron silicate.
- 78. Method according to any of claims 67-77, wherein the compound is selected from and comprisies any combination from the group of: metal oxide, semi-metal oxide, metal oxidhydroxide, semi-metal oxidhydroxide, metal hydroxide, semi-metal hydroxide, metal carbide, semi-metal carbide, metal nitride, semi-metal nitride, metal carbonitride, semi-metal boride identical to at least one of a number of compounds resulting from the formation in said reactor.
 - 79. Method according to any of claims 1-78, wherein the solid reactor filling material comprises a ceramic.

- 80. Method according to any of claims 1-79, wherein the solid reactor filling material comprises a metal sulphate.
- 81. Method according to any of claims 1-80, wherein the solid reactor filling material comprises a metal halide.
 - 82. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a metal oxide, a metal oxidhydroxide or a metal hydroxide identical to said compound resulting from the formation in said reactor.

10

- 83. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a semi-metal oxide, a semi-metal oxidhydroxide or a semi-metal hydroxide identical to said compound resulting from the formation in said reactor.
- 15 84. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a metal carbide identical to said compound resulting from the formation in said reactor.
- 85. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a semi-metal carbide identical to said compound resulting from the formation in said reactor.
- 86. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a metal nitride identical to said compound resulting from the formation in said reactor.
 - 87. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a semi-metal nitride identical to said compound resulting from the formation in said reactor.

- 88. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a metal carbonitride identical to said compound resulting from the formation in said reactor.
- 35 89. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a semi-metal carbonitride identical to said compound resulting from the formation in said reactor.

- 90. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a metal boride identical to said compound resulting from the formation in said reactor.
- 5 91. Method according to any of claims 1-81, wherein the solid reactor filling material comprises a semi-metal boride identical to said compound resulting from the formation in said reactor.
- 92. Method according to any of claims 1-91, wherein the solid reactor filling material comprises any combination of metal oxide, semi-metal oxide, metal oxidhydroxide, semi-metal oxidhydroxide, metal hydroxide, semi-metal hydroxide, metal carbide, semi-metal carbide, metal carbonitride, metal carbonitride, metal boride and semi-metal boride identical to at least one of a number of compounds resulting from the formation in said reactor.

15

- 93. Method according to any of claims 1-92, wherein the solid reactor filling material functions as seed material for the formation of said compound.
- 94. Method according to any of claims 1-92, wherein the solid reactor filling material functions as a collecting agent for the said compound.
 - 95. Method according to any of claims 1-94 wherein said compound is separable from the solid reactor filling material with no further treatments of the solid reactor filling material.
- 96. Method according to any of claims 1-95, wherein said compound is separable from the solid reactor filling material without substantially degrading the solid reactor filling material.
- 97. Method according to any if claims 1-96, wherein said compound is separable from the solid reactor filling material in a way that allows the solid reactor filling material to be reused as solid reactor filling material.
 - 98. Method according to any of claims 1-97, wherein said compound is separable from the solid reactor filling material by flushing the solid reactor filling material in a fluid.

35

99. Method according to any of claims 1-98, wherein said compound is separable from the solid reactor filling material by vacuum means.

100. Method according to any of claims 1-99, wherein said compound is separable from the solid reactor filling material by blowing means.

- 101. Method according to any of claims 1-100, wherein said compound is separable from5 the solid reactor filling material by ultrasonic means.
- 102. Metal compound such as metal oxide, metal oxidhydroxide, metal hydroxide, metal carbide, metal nitride, metal carbonitride or metal boride compound being manufactured by the method according to any of claims 1-101, wherein the metal oxide, metal
 10 oxidhydroxide, metal hydroxide, metal carbide, metal nitride, metal carbonitride or metal boride compound is in the form of aggregates of primary particles with an average primary particle size of maximum 1000 nm, preferably maximum 500 nm, and most preferably maximum 100 nm.
- 15 103. Metal compound such as metal oxide, metal oxidhydroxide, metal hydroxide, metal carbide, metal nitride, metal carbonitride or metal boride compound being manufactured by the method according to any of claims 1-101, wherein the metal oxide, metal oxidhydroxide, metal hydroxide, metal carbide, metal nitride, metal carbonitride or metal boride compound is in the form of aggregates of primary particles with an average primary particle size of 100 nm, preferably maximum 50 nm, more preferably maximum 20 nm, and most preferably maximum 10 nm.
- 104. Semi-metal compound such as semi-metal oxide, semi-metal oxidhydroxide, semi-metal hydroxide, semi-metal carbide, semi-metal nitride, semi-metal carbonitride or semi-metal boride compound being manufactured by the method according to any of claims 1-101, wherein the semi-metal oxide, semi-metal oxidhydroxide, semi-metal hydroxide, semi-metal carbide, semi-metal nitride, semi-metal carbonitride or semi-metal boride compound is in the form of aggregates of primary particles with an average primary particle size of maximum 1000 nm, preferably maximum 500 nm, and most preferably
 30 maximum 100 nm.
- 105. Semi-metal compound such as semi-metal oxide, semi-metal oxidhydroxide, semi-metal hydroxide, semi-metal carbide, semi-metal nitride, semi-metal carbonitride or semi-metal boride compound being manufactured by the method according to any of claims 1101, wherein the semi-metal oxide, semi-metal oxidhydroxide, semi-metal hydroxide, semi-metal carbide, semi-metal nitride, semi-metal carbonitride or semi-metal boride compound is in the form of aggregates of primary particles with an average primary particle size of 100 nm, preferably maximum 50 nm, more preferably maximum 20 nm, and most preferably maximum 10 nm.

- 106. Metal compound being manufactured by the method according to any of claims 1-101, wherein the metal compound is a metal oxide product being TiO2, preferably with a crystallinity of minimum 20%, preferably minimum 30%, more preferably minimum 40%, 5 and even more preferably minimum 60% and even most preferably minimum 80%.
 - 107. Metal compound being manufactured by the method according to any of claims 1-101, wherein the metal compound is a metal oxide product being TiO₂ of anatase structure.
- 10 108. Metal oxide product being manufactured by the method according to any of claims 1-101, wherein the metal compound is a metal oxide being from the group of: Al₂O₃, TiO₂, ZrO_2 , Y_2O_3 , WO_3 , Nb_2O_5 , TaO_3 , CuO, CoO, NiO, SiO_2 , Fe_2O_3 and ZnO.
- 109. Metal compound being a metal oxidhydroxide compound being manufactured by the 15 method according to any of claims 1-101, wherein the metal oxidhydroxide is from the group of: iron oxidhydroxide, titanium oxidehydroxide, manganese oxidhydroxide and aluminium oxidhydroxide.
- 110. Metal compound being a metal oxidhydroxide compound being manufactured by the 20 method according to any of claims 1-101, wherein the metal oxidhydroxide is aluminium oxidhydroxide of Boehmite structure.
- 111. Metal compound being a metal hydroxide compound being manufactured by the method according to any of claims 1-101, wherein the metal hydroxide is from the group 25 of: iron hydroxide, silicon hydroxide, zirconium hydroxide, titanium hydroxide, manganese hydroxide and aluminium hydroxide.
- 112. Apparatus for manufacturing a metal compound such as metal oxides, metaloxy hydroxides metal hydroxides, metal carbides, metal nitrides, metal carbonitrides, metal 30 borides, electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the following components:
 - means for introducing a solid reactor filling material in a reactor,
 - means for introducing a metal-containing precursor in said reactor,
 - means for introducing a reactant in said reactor,
- 35 means for introducing a supercritical solvent into the said reactor,
 - said reactor intended as a space for establishing a contact between the metal-containing precursor and the reactant and
 - said reactor intended as a space for the formation of said compound in the proximity of the said solid reactor filling material.

following components:

- 113. Apparatus for manufacturing a semi-metal compound such as semi-metal oxides, semi-metaloxy hydroxides semi-metal hydroxides, semi-metal carbides, semi-metal nitrides, semi-metal carbonitrides, semi-metal borides, electroceramics and other such 5 compound, said compound having a sub-micron primary particle size, comprising the
 - means for introducing a solid reactor filling material in a reactor,
 - means for introducing a semi-metal-containing precursor in said reactor,
 - means for introducing a reactant in said reactor,
- 10 means for introducing a supercritical solvent into the said reactor,
 - said reactor intended as a space for establishing a contact between the semi-metalcontaining precursor and the reactant and
 - said reactor intended as a space for the formation of said compound in the proximity of the said solid reactor filling material.

- 114. Apparatus for manufacturing a metal compound such as metal oxides, metaloxy hydroxides metal hydroxides, metal carbides, metal nitrides, metal carbonitrides, metal borides, electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the following components:
- 20 means for introducing a solid reactor filling material in a reactor,
 - means for introducing a metal-containing oxide in said reactor,
 - means for introducing a substitution source in said reactor,
 - means for introducing a supercritical solvent into the said reactor,
 - said reactor intended as a space for establishing a contact between the metal-containing
- 25 oxide and the substitution source and
 - said reactor intended as a space for the formation of said compound in the proximity of the said solid reactor filling material.
- 115. Apparatus for manufacturing a semi-metal compound such as semi-metal oxides, 30 semi-metaloxy hydroxides semi-metal hydroxides, semi-metal carbides, semi-metal nitrides, semi-metal carbonitrides, semi-metal borides, electroceramics and other such compound, said compound having a sub-micron primary particle size, comprising the following components:
 - means for introducing a solid reactor filling material in a reactor,
- 35 means for introducing a semi-metal-containing oxide in said reactor,
 - means for introducing a substitution source in said reactor,
 - means for introducing a supercritical solvent into the said reactor,
 - said reactor intended as a space for establishing a contact between the semi-metalcontaining oxide and the substitution source and

- said reactor intended as a space for the formation of said compound in the proximity of the said solid reactor filling material.